Synthesis Characterization and Physical Property of Activated Porous Carbon and its Metal Oxide Composite for Energy Storage Application

Final Synopsis submitted to fulfillment of the requirements for the degree of

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in

P H Y S I C S

Submitted By

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Synopsis

1. Introduction

The growing of modern digitalization, population growth, and industrial demands are created a scarcity of raw material is the one of present challenge of the researchers to identify new sustainable material for energy storage and conversion devices with flexible size, design, and alternative energy storage technologies that are cost-effective, environmentally friendly are important to decreasing fossil fuel consumption and reducing environmental pollution [1]. The advanced new scientific technology mainly in the direction of portable devices, computerization, and internet appliances are focused area in energy consumption necessitates to exploration of storage sources. To enhance the viability and success of these eco-friendly source material, the finding of an appropriate new energy materials is very essential [2]. In recent years, the development of sustainable energy conversion and storage materials derived from biomass and bio-waste, with the aim of mitigating environmental issues, has garnered significant attention [3]. The supercapacitors, a type of energy storage devices with a high power density, short charging-discharging times, and ultra-long cycle life, have attracted a lot of attention recently due to their numerous uses in consumer electronics, electric vehicles, renewable energy sources, and uninterruptible power supplies [4]. The carbon nanotubes and graphene have been widely employed as electrode materials in electric double-layer capacitors (EDLCs) owing to their eco-friendliness and superior electrical conductivity, but, their high costs have made them limited in application on a large scale [5]. Activated carbon are major active materials used in EDLC because of their large specific surface area (SSA), adjustable pore size, medium electrical conductivity and good electrochemical stability [6]. The pore

structure of the activated carbon has a significant influence on their electrochemical performances during the redox mechanism.

On the other hand, many natural materials are generally abundant, renewable, inexpensive, and environmentally stable compared to artificial templates and precursors, as is well known, with the development of industry and the economy. There is a huge bio-waste product generated domestically which is becoming a challenge to handle and also environ mental pollution; thus, as an alternative, recycling bio-waste or converting it into useful industrial products is crucial in preserving the environment [7]. Biomass waste contains high proportions of carbohydrates such as cellulose and lignin, which can be used to develop cheap activated carbon. The procedure for the fabrication of activated carbons (ACs) from biomass precursors is simple and more environmentally friendly [8, 9]. Moreover, biomass-derived ACs, which have been used in several industrial applications due to unique properties such as ultrahigh surface area, tailorable pore size, low toxicity, good electrical conductivity, high chemical stability, and natural presence of desirable surface oxygen functional groups [10]. Appropriate specific surface area and pore size distribution are the key factors that decide the storage capacity of carbon materials. The appearance of hierarchical porous carbons (HPCs) facilitated the development of novel structures, establishing it as an effective electrode material for supercapacitors due to its three-dimensional (3D) porous channels, including a well-balanced micro-, meso-, and macroporous structure [11]. The HPCs have significant porosity characterized by well-interconnected pores, comprising micropores less than 2 nm, mesopores ranging from 2 to 50 nm in diameter, and macropores above 50 nm in size. Compared to ordered mesoporous carbons and activated carbons, which have low capacitance and poor rate

capability, this kind of micro-/nanostructured carbon has shown better electrochemical performance [12].

Recently, the investigations of composite for achieving synergistic properties between EDLCs and pseudocapacitors are emerging research to resolve energy demand by bringing alternative energy storage system. The joint properties of EDLCs and pseudocapacitors show promising potential for developing high-performing and stable new electrode materials through the combination of porous carbons with pseudocapacitive materials. The transition metal oxides such as NiO [13, 14], Co_3O_4 [15, 16], ZnO [17, 18], and CuO [19, 20], represent pseudocapacitive materials that exhibit high energy density along with large capacitance while offering simple transition pathways to nanoparticle form for surface integration with carbon materials [21, 22]. Cobalt oxide has been demonstrated as a viable pseudo-capacitive metal oxide additive due to its various stable oxidation states (Co^{2+}, Co^{3+}, and Co^{4+}), low cost, non-noble nature, and high theoretical specific capacitance [23]. Which could be incorporated on the porous carbon for obtaining high-performance supercapacitors. The synergistic impact of the three-dimensional porous carbon and cobalt oxide nanoparticles endows the composite electrode with superior electrochemical characteristics, owing to the high specific surface area of the material, which enhances the ion response to the active sites. Yuanchuan et al. used carbonized butterfly wing scales as the electrode to grow Co_3O_4 nano-arrays to form N-doped, multichannel, hierarchically porous, and 3D hierarchically periodic structures to offer high performance for the electrochemical capacitors [24]. Yajun et al. synthesized multi-heteroatom doped porous carbons, which were derived from agricultural waste as support material. Next, cobalt oxide nanosheets were deposited on the surface of the carbon material to achieve the

composites. The composite electrode materials possess high specific surface area with low electrochemical impedance [25].

Nickel oxide (NiO) is also an effective supercapacitor material. It has a large surface area, is non-toxic, and is simple to produce, with a large 2584 F/g capacitance [26]. NiO can be developed into a variety of nanostructures, including nanoflakes, nanosheets, nanoribbons, and nanowires, employing microwave-assisted, hydrothermal/solvothermal, and electrodeposition processes[27]. Shuyang et al. synthesized honeycomb NiO nanosheet @ wheat husk-based porous carbon framework (NiO@PC). The high surface area of porous carbon (634.4 $m^2 g^{-1}$) enhanced the bonding of NiO nanosheets, and this hierarchical nanostructure (NiO@PC) facilitated electron and electrolyte ion transport while accelerating the reversible redox reaction, thereby effectively demonstrating its superior energy storage capabilities as a supercapacitor [28]. Walnut shell-derived activated carbon altered with nickel oxide (NiO) nanoparticles exhibited an enhancement in the specific capacitance of the supercapacitor electrode by 16% in comparison to the original unmodified activated carbon [29].

The CuO has attractive properties such as easy synthesis, non-toxicity, economic viability, and outstanding electrical, optical, and electrochemical properties. These characteristics have caused scholarly interest in CuO as electrode for supercapacitors. CuO can produce a higher theoretical value of specific capacitance, up to 1800 F/g [19]. The addition of copper oxide nanoparticles reduces the electron transport and ion diffusion obstacles of porous carbon substrates, resulting in increased specific capacitance of the electrode materials. Yinbo Zhan et al. prepared biomass-derived carbon materials embedded with CuO nanoparticles with the help of cheap peanut shells as a carbon source and copper acetate as the CuO source. The synthesized CuO-AC material retains a large BET surface area of 2640.55 $m^2 g^{-1}$ with micro-mesoporous

structures in a hierarchical manner. The CuO-AC electrode shows a specific capacitance of 530 F/g at 1 A/g [30]. Fuming et al. prepared Cu_2O-loaded S-doped worm-like hierarchical porous carbon flakes (SCu-HPCFs) for supercapacitor. The as-prepared SCu-HPCFs possess hierarchical porous structure, large specific surface area (2543.68 m^2 g^{-1}), s doping state and good elec- trical conductivity [31].

Zinc oxide (ZnO) has been considered one of the most promising transition metal oxide materials for the construction of pseudocapacitor because of its low cost, eco-friendliness and simple synthesis[32]. This material is widely used in solar cells, gas sensors, and short-wavelength light-emitting appliances because of its excellent optoelectronic characteristics [33]. It has also been given special consideration as an electrode material due to its battery-active material and high energy density of 650 Ag^{-1} [34]. However, it has the disadvantage of reducing the cycle life of the electrode since dendritic growth develops in subsequent cycles. Nevertheless, these limitations can be overcome by using several types of composite materials made of carbon and zinc oxide, which are used as electrodes in supercapacitors. Hence, the integration of metallic oxides into carbon materials is anticipated to enhance the utilization of both the faradaic capacity of the metallic oxide and the dual-layer capacity of the carbon materials with extensive specific surface areas, thereby improving the capacity and energy/power potential [35].Moreover, in the absence of a support structure, metal oxide structures typically exhibit agglomeration, which notably reduces their electrochemical performance. Recent investigations have intensely focused on hybrid systems that combine carbon materials with metal oxides to achieve synergetic features, characterized by high energy and power densities alongside robust cycle stability [36].

The review of literature on energy storage materials reveals a more diversed strategy on the fabrication of design and facile technology with cost-effective, sustainable, and eco-friendly energy storage solutions. Present investigation is focused on the fabrication of activated carbon produced from biomasses and composited with metal oxide. In other words, abundant waste biomasses can be transformed into valuable energy materials for energy storage applications.

2. Aims and objective of the present research work

The main objectives of the studies to develop low cost, environment friendly, high-performance based activated porous carbon composite electrodes for electrochemical capacitors, which is described as follows:

i. To synthesis activated porous carbon using agro-waste material as carbon source, and also with metal oxide composite using chemical/hydrothermal methods.
ii. To characterize activated porous carbon and its composite using different techniques such as FT-IR, XRD, HRTEM, FESEM, TGA, BET, and Raman spectroscopy to know the chemical, structural, morphology, and microstructural arrangement.
iii. To study the electrochemical properties such as cyclic voltammetry (CV), Galvanostatic charge–discharge (GCD), and Electrochemical Spectroscopy, the synthesized porous carbon composite electrode for supercapacitor applications.

3. Outcome of Research work

The proposed research work discussed on the electrochemical double-layer capacitors. It providing the comprehensive experimental and theoretical interpretation of environmental sustainable device material and the working mechanism of electrochemical double-layer capacitors. The fabricated device demonstrates a superior specific capacitance between 400 -

800 F/g and a high energy density of (40-130) Wh kg^{-1}. These results are suggesting that activated porous carbon with a transition metal oxide composite is remarkably favorable in supercapacitor applications**.**

4. Brief outline of the proposed thesis

The proposed thesis is divided into seven chapters; the first chapter contains the introduction of materials and their properties as well as activated porous carbon and transition metal oxides with their properties. The second chapter deals with the synthesis method and characterization techniques to employ the synthesized activated porous carbon with transition metal oxide composite and their fabrication process. The third to sixth chapters are working chapters that provides the result and discussion of observed results. The seventh chapter covered conclusion and scope for the future work.

Chapter-I: Introduction

This chapter elaborates on the broad information about the introduction of materials, electrochemical double-layer capacitor mechanism, carbon-based materials, and metal oxide composite properties, and analyzes species. Literature review for related topic, motivation, aims, and objectives of the work described in detail.

Chapter-II: Experimental and Characterization Techniques

This chapter describes the synthesis methods of activated porous carbon and transition metal oxides and their characterization to analyze the morphology, structural modification, elemental identification, electrode fabrication, and electrochemical properties.

Chapter-III: Facile synthesis of bio-waste date seed derived hierarchical porous carbon/cobalt oxide composite for supercapacitor applications

In this chapter, it is demonstrated that a facile and sustainable method for synthesizing a bio-waste date seeds-derived porous carbon cobalt oxide composite by a facile one-step carbonization and chemical activation process using KOH as an active agent. The morphology of the composite demonstrated a hierarchical high porous carbon with cobalt oxide uniform distributed on the surface of porous carbon network. The obtained PCCo composite has a porous structure with carbon frameworks and uniformly dispersed Co_3O_4 nanoparticles. It is observed a high specific surface area (397 m^2 g^{-1}), and abundant mesopores. This hierarchical architecture offers good ion/electron transport channels for better electrochemical characteristics. The maximum specific capacitance was found to be 496 F g^{-1} at a scan rate of 10 mV/s, and also from the galvanostatic charge-discharge curve, it was 671 F g^{-1} at a current density of 1.5 A g^{-1}. The fabricated SSC PCCo-0.4//PCCo-0.4 device provides an energy density of 47.4 Wh kg^{-1} and a power density of 853.2 W kg^{-1} with capacitance retention of 84.4% and coulombic efficiency of 97% even after 5000 cycles. Therefore, these unique properties enable the material to become a promising high-performance electrode material for supercapacitors. Moreover, it addresses the problem of bio-waste recycling and provides a method for generating advanced materials for energy storage at the industrial level.

Chapter-IV: Fabrication of high-performance supercapacitor electrode from coconut shell-derived activated carbon -nickel oxide nanocomposite

This chapter described a cost-effective and environmentally friendly method for preparing activated porous carbon derived from coconut shells incorporated nickel oxide nanocomposites (PCNiO) through a two-step carbonization followed by a hydrothermal process. The PCNiO nanocomposites show roughness with a high porous structure due to nickel ions dispersed on the surface. The electrochemical properties of the PCNiO nanocomposite electrodes were tested using a two-electrode system. At a current density of 1.5 A g^{-1} the GCD revealed that the Csp of the PCNiO electrode was 656.2 F g^{-1}. According to the CV curve, the Csp range was 598.6 F g^{-1} at a scan rate of 10 mV/s. Additionally, even after 5000 cycles, the electrode had a coulombic efficiency of 97.55 % and a strong cycle stability, with a capacitance retention of 78.34%. The PCNiO electrode also demonstrates a high ED of 44 Wh kg^{-1} and a PD of 562.5 W kg^{-1}. These results indicate that the integration of nickel oxide into activated porous carbon demonstrates significant improvements in specific capacitance, which can be ascribed to its increased surface area and enhanced ion diffusion rates.

Chapter-V: Synthesis of cantaloupe peel-derived 3D-interconnected porous carbon-copper oxide nanocomposite for high-performance symmetrical supercapacitor

In this chapter, a novel approach to synthesizing 3D-interconnected porous carbon material derived from cantaloupe peel is presented, integrated with copper oxide nanoparticles (PCCuO) by using one-step carbonized activation for enhanced symmetric supercapacitor performance (SSC). The structural and morphological analyses reveal a well-defined porous structure with

uniformly dispersed copper oxide nanoparticles. Owing to a 3D-interconnected porous structure and high specific surface area (307.29 m^2 g^{-1}), PCCuO-0.25 electrode demonstrates exceptional electrochemical performance, achieving a specific capacitance (Csp) of 673 F g^{-1} at a current density (CD) of 1.5 A g^{-1} in 6 M KOH electrolyte. A symmetric supercapacitor (SSC) utilizing PCCuO-0.25 for both positive and negative electrodes, along with a PVA/KOH gel electrolyte, attains a maximum energy density (ED) of 55 Wh kg^{-1} at a power density (PD) of 636 W kg^{-1}, showing outstanding cycle stability with capacitance retention of 81% and coulombic efficiency of 98.2% after 5000 charging–discharging cycles. With our work, the main developments and importance of applying the findings are that we have highlighted the capability of renewable biomass resources, treated sustainability issues, and minimized the environmental issues in relation to electrode material synthesis.

Chapter-VI: Developing hierarchical porous Carbon-Zinc oxide nanocomposite electrode for supercapacitor applications

This chapter described a new approach for the synthesis of hierarchical porous carbon/zinc oxide (ZnO) nanocomposite (HPCZ) with excellent electrochemical performance for symmetric supercapacitors. The hierarchical porous carbon/ZnO nanocomposite (HPCZ) was synthesized through a simple hydrothermal treatment followed by a carbonization process; the date seeds are used as a precursor, and ZnO nanoparticles (NPs) are incorporated at the same time. The resulting HPCZ nanocomposite revealed a 3D interconnected porous structure with ZnO NPs homogeneously dispersed to ensure that the ion transport channels from the bulk electrolyte to the active sites in the porous matrix are effectively provided. The fabricated nanocomposites HPCZ provided prominent specific capacitance of 707.9 F g^{-1} at a CD of 2 A

g^{-1}, with relatively high energy density of 125.5 Wh kg^{-1} at 1129.5 W kg^{-1}, especially with a small amount of the ZnO NPs, and exhibited appropriate specific energy and high power density owing to the synergistic effect between carbon materials and ZnO. It's also exhibited remarkable cycling performance with retention of up to approximately 92.2%, and coulombic efficiency is 98% over 5000 cycles. The facile synthesis method and use of abundant, low-cost date seeds as a carbon source make this HPCZ nanocomposite system attractive for scalable, cost-effective supercapacitor fabrication.

Chapter-VII: Conclusions and Scope for future work

This chapter presents the conclusions of obtained results and the scope for future work.

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List of Journal Publications and Conference Proceedings

[1] A paper entitled "Bio-waste-derived one-step carbonized hierarchical structured carbon/ cobalt oxide composite for energy storage application" in Ionics journal Springer Publication, Febraury 2025.
Abdullah Ba shbil, Nagaraju Y S, Ganesha H, Veeresh S, Suresh D S, Vijaykumar S P, Sapna Sharanappa, Devendrappa H. https://doi.org/10.1007/s11581-025-06072-z

[2] A Paper entitled "Nilgiri tree peels-derived activated porous carbon for high performance supercapacitor applications" published in IOP Conf. Ser.: Mater. Sci. Eng. 1300 (2024) 012010.
Abdullah Ba shbil, Nagaraju Y S, Ganesha H, Veeresh S, Suresh D S, Vijaykumar S P, Sapna Sharanappa. doi:10.1088/1757-899X/1300/1/012010

[3] A paper entitled "Sustainable approach to fabricate high performance symmetry supercapacitor electrode from natural coconut shell-derived porous activated carbon with nickel oxide nanocomposite" Accepted, in ACS Omega Journal, February 2025.
Abdullah Ba shbil, Nagaraju Yennappa Siddappa, Suresh Daddi Suraiah, Ganesha Honnu, Vijaykumar Siddappa Pujar, Sapna Sharanappa, Devendrappa Hundekal *.

Conference/Workshop attended

[1] Participated and presented a paper entitled "Nilgiri tree peels-derived activated porous carbon for high performance supercapacitor applications" in the International Conference on Physics of materials and Nanotechnology (ICPN-2023) organized by Department of Physics, Mangalore University held from 21st - 23rd September, 2023.
Abdullah Ba shbil, Nagaraju Y S, Ganesha H, Veeresh S, Suresh D S, Vijaykumar S P, Sapna Sharanappa.

[2] Participated in the workshop on "Research Publication & Ethics" on 18 – 23rd February 2022 at Department of Education, Mangalore University, Mangalagangothri.

[3] Participated in the "Basic Training Program in Nano Science and Technology", supported by Ministry of Education (MoE), Govt. of India and conducted by the Centre for Nano Science and Engineering, Indian Institute of Science, Bangalore, from 20 – 22 June 2022.

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